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Solventless Method for Determining Moisture Content of Solid Propellants

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13. ABSTRACT (Maximum 200 words) A new method to determine moisture content of solid propellants has been developed. The method is based on mass spectrometric detection of thermally desorbed water. The main advantages of this method over existing methods is that sample preparation is very simple and no extraction with dry solvents is required. Furthermore, analysis can be performed even if only a very small quantity of a sample is available. The two main disadvantages of the method are that special instrumentation is required, and that multiple analyses must be performed to obtain representative results (since only small samples can be examined). Comparison with conventional methods of moisture analysis have not yet been performed.				
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1. INTRODUCTION

There are several methods that may be used to determine moisture content of solid propellants. Gravimetric techniques involve heating the propellant and monitoring weight loss. For single-base propellants, this is a simple matter. For double- and triple-base propellants, the presence of volatile plasticizers such as nitroglycerine (NG) and diethylene-glycol dinitrate (DEGDN) can complicate the determination. Other methods (i.e., MIL-STD-2668 and JANNAF 523.1) involve first extracting water with dry solvents, and then analyzing the extract by liquid or gas chromatography. Using these methods, extraction times can last as long as 16 hr; consumption of 50 mL of solvent per sample is not unusual. Furthermore, local environmental conditions such as high ambient humidity can make it difficult to keep solvents and glassware dry. Methods based on Karl Fisher titrations also require the use of dry solvents, reagents, and glassware. The disadvantages of these techniques are that they are time-consuming, require the use of volatile organic compounds (VOCs), and generate reactive hazardous waste.

An alternate method for moisture determination has recently been developed in response to an urgent request for analysis of JA2 samples suspected to have been exposed to excessive moisture. Conventional extraction methods could not be employed because there was neither time to dry the required solvents (MIL-STD-2868 recommends that solvents remain over molecular sieves for a minimum of two days before use) nor the local environmental conditions to keep the solvents dry (due to high ambient humidity and the absence of adequate air conditioning). To meet the suspense for the required analyses, it was decided that an alternate, solvent-free technique must be developed. This was successfully accomplished, and yielded results in a relatively short time (instrument calibration plus approximately 30 sample runs in 10 hr). In addition, the method did not require the use of VOCs and consumed only small amounts of propellant. Considering the high cost of disposal for reactive hazardous wastes, this resulted in a significant savings of both time and money.

The main disadvantages of the method are that 1) multiple analyses are required to assure representative results (since the test requires a very small sample size), and 2) the method required specialized instrumentation (e.g., a device in which materials may be desorbed from the propellant and then transferred directly into a gas chromatograph).

2. EXPERIMENTAL

2.1 Instrumentation. Moisture desorption was achieved via a CDS Model 122 Pyroprobe (coil type) connected to a heated interface chamber to the splitless injector of a Hewlett Packard GC-FTIR-MS system (Model 5890 GC, Model 5970 MSD, and Model 5965 IRD with narrow band MCT detector). The GC column used was a Quadrex capillary column (0.32 mm \times 25 m; 3 μ m OV-17 film). The injector temperature was 200° C. A 200° C isothermal GC program was used.

2.2 Procedure. Six JA2 samples (approximately 5 g each) were provided for chemical analysis (see Table 1 for sample description). A seventh sample of JA2 was also analyzed for comparative purposes. Using a razor blade, cross-sectional slices (<1 mm thickness) of the solid propellant were cut. Cross-sectional slices were then cut into strips (<1 mm diameter) and placed into preweighed quartz tubes containing a plug of glass wool. The glass wool was used to prevent propellant from coming out of the tube. Quartz tubes containing the propellant were then reweighed to determine propellant mass.

Table 1. Designation for JA2 Samples Analyzed

Sample	Lot No.	Designation
19 perf, Stick, Sample A	HCL94A015-002	A-19
19 perf, Stick, Sample B	HCL94A015-002	B-19
7 perf, Stick, Sample A	HCL93J014-001	A-7
7 perf, Stick, Sample B	HCL93J014-001	B-7
7 perf, Granular, Sample A	HCL93E-071425	A-G-7
7 perf, Granular, Sample B	HCL93E-071425	B-G-7
Unperforated, Stick, "STD"	RAD-PDI-002-1F	JA2-stk

The quartz tube containing the propellant was then placed within the coils of the pyroprobe heating element (see diagram in Figure 1), which was subsequently inserted into the pyroprobe interface and screwed into place. At the start of the GC run, a 150° C pulse (20-s duration) was given to the sample via the pyroprobe. It was confirmed that these conditions are sufficient for desorption of all moisture by giving a second pulse to the sample, and observing no subsequent moisture desorption. In preliminary

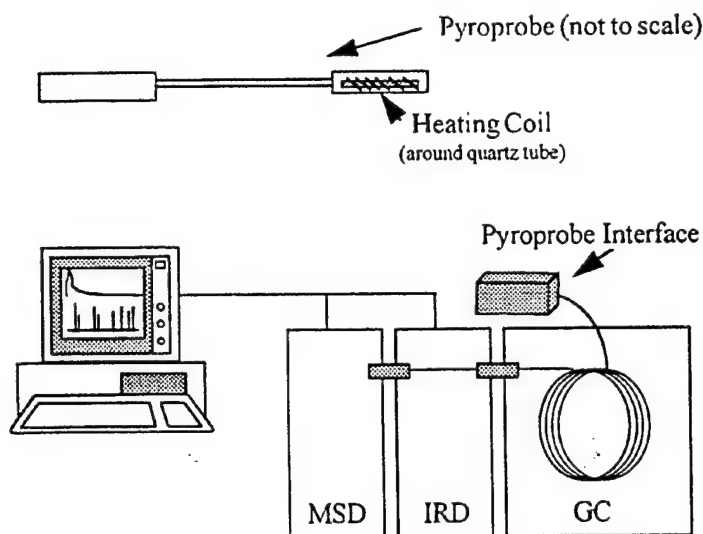


Figure 1. Schematic representation of experimental apparatus.

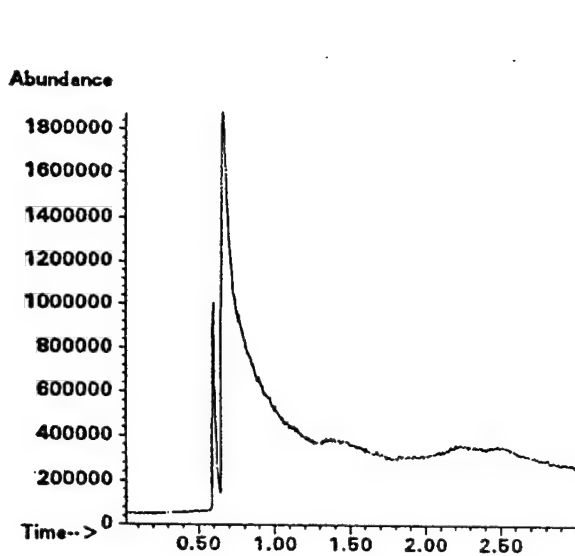
studies it was noted that if the sample size was too large (i.e., above 30 mg) or the propellant slices were packed too closely in the tube, it was difficult to desorb all the moisture with just one pulse.

For preparation of a calibration curve, aliquots (0.1–0.8 μL) of water were transferred to a plug of glass wool in a quartz tube, and then analyzed as previously described.

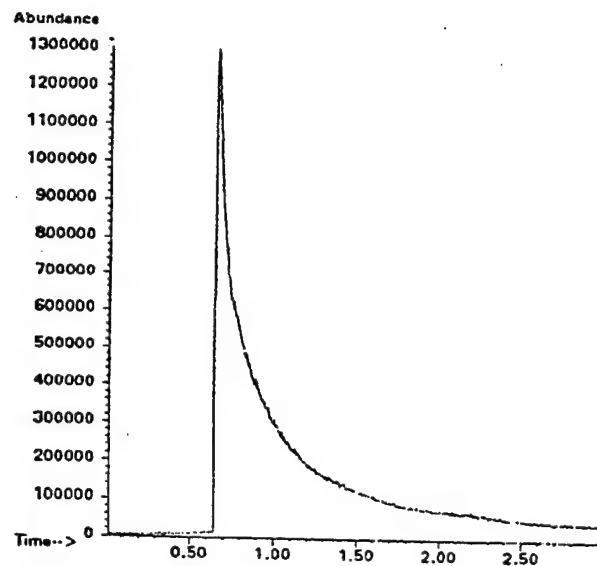
Gas chromatograms, total ion chromatograms (TICs) (based on MS response), and total response chromatograms (TRCs) (based on IR response) were collected. For the purpose of this analysis, only the TICs were necessary. Selected ion chromatograms (SICs) were also obtained to distinguish between response due to desorbed water and plasticizer (the peaks overlap with one another). Integration of the $m/z = 18$ SIC yielded the peak area for water. An example of a TIC and SIC are given in Figure 2.

3. RESULTS

3.1 Instrument Calibration. The calibration curve obtained by analysis of known volumes of water is given in Figure 3 ($R^2 = 0.995$).



(a) Total Ion Chromatogram



(b) Selected Ion Chromatogram
($m/z = 18$, water)

Figure 2. Gas chromatograms.

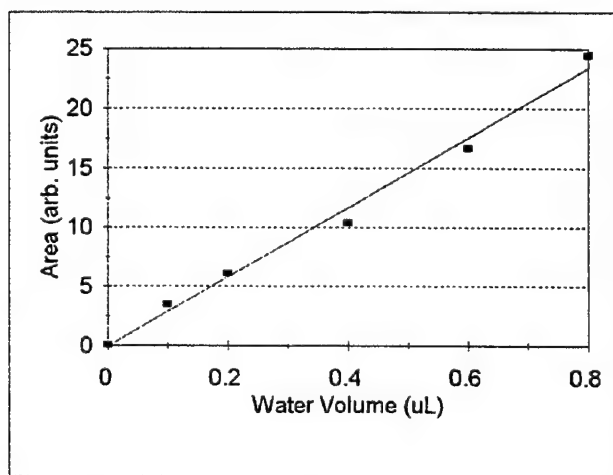


Figure 3. Calibration curve for moisture analysis.

3.2 Moisture Content. Table 2 gives the results for moisture analysis. Given that the specifications for JA2 propellant call for 0.5 ± 0.3 weight-percent moisture, it is concluded that all seven samples fall within the limit of 0.2–0.8 weight-percent.

Table 2. Results for Moisture Analysis of JA2 Propellant

Sample ID	Peak Area (arb units)	Mass (mg)	Water (μ L)	Water (weight-percent)
A-19	2.85	19.0	0.127	0.67
A-19	2.17	16.3	0.104	0.64
B-19	2.53	19.7	0.116	0.59
B-19	3.35	24.3	0.144	0.59
A-7	3.23	24.5	0.140	0.57
A-7	3.83	27.4	0.160	0.58
B-7	3.50	24.0	0.148	0.62
B-7	2.94	19.3	0.126	0.67
A-G	2.82	29.2	0.126	0.43
A-G	3.24	26.7	0.140	0.52
B-G	2.43	17.5	0.113	0.64
B-G	1.55	11.2	0.081	0.74
JA2-Stk	1.71	13.4	0.088	0.66
JA2-Stk	2.23	20.0	0.106	0.53

3.3 JA2 Soaked in Water. To confirm that JA2 grains would not absorb excessive moisture even if soaked in water, a stick of 7-perf stick (Sample A) was soaked overnight at room temperature in filtered water. Prior to analysis, the stick was patted dry with a paper towel and then analyzed as previously described. No significant increase in moisture level was observed.

4. CONCLUSIONS

To meet the deadline on a "short-suspense" analysis of JA2 propellant, a new solvent-free analytical method for moisture determination was developed. Although the method was not validated by comparison with traditional methods, it did provide what appear to be reasonable results for several JA2 samples. The method is quick, consumes little propellant, and does not require the use of solvents. However, it does

require special instrumentation and multiple analyses (due to limitations on sample size). It is recommended that this method be run side-by-side with traditional methods to confirm that its results are reliable and to demonstrate applicability to other propellant samples.

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